

# Er strengthening aluminum alloy

## 1. Cross reference to related applications

The present application claims the benefit of Chinese applications CN 03119119.3, filed on March 14, 2003, entitled “Al-Zn-Mg-Er series rare earth element containing aluminum alloy” and CN 03153576.3, filed on August 18, 2003, entitled “Al-Mg-Li-Zr-Er series alloy”, both of which are herein incorporated by reference in their entirety.

## 2. Background of the invention

### 2.1 Field of the invention

The present invention relates to aluminum alloys, specifically to Er-containing aluminum alloys for structural purposes, e.g., Al-Zn-Mg-Er alloys, Al-Mg-Er alloys and Al-Mg-Li-Zr-Er alloys.

### 2.2 related prior arts

Since aluminum alloy has good specific strength and corrosion resistance, it has been widely used in various fields. However, with the development of the technology, now a need has arisen to further improve the performance of aluminum alloy especially the mechanical properties. In this connection, various researches have been conducted to improve the mechanical performance of aluminum alloy by alloying such by addition of rare earth elements, for instance, Meng Liang et al. in the paper “Effects of Ce and Impurities on Percentage Elongation and Recrystallization in Al-Li Alloys”, Journal of Rare Earths, vol.16[2],1998, disclosed the effect of Ce on the percentage elongation and recrystallization behavior in Al-Li Alloys and found the elongation was increased by Ce micro-alloying. To the knowledge of related researches , among the rare earth alloying elements, Sc is up to now regarded as the most effective micro-alloying element to

increase the mechanical properties of aluminum and aluminum alloys. However, Sc is much expensive and brings about larger increase of the production cost. Therefore, a need still exists to find out more cost-efficient aluminum alloys with high mechanical properties.

The present applicant surprisingly found that despite the substantial differences among the various aluminum alloys, the addition of Er into the aluminum alloys could greatly increase the strength without significant deterioration of the toughness. Furthermore, Er is much cheaper than Sc etc., so that the addition of Er can improve aluminum alloys with both high mechanical properties and more cost efficiency.

Al-Zn-Mg series alloys are typical commercial aluminum alloys, widely used in such applications as aerospace, aircrafts, and transportations.

The applicant found that beneficial effects such as markedly refined grain size and significant increase in strength could be obtained by the addition of Er (erbium) into Al-Zn-Mg series alloys that can be strengthened by heat treatment (e.g. the aluminum alloy comprises Zn5.0 ~ 7.0Wt%, Mg1.5 ~ 2.5Wt%, the balance being Al), and similar advantageous effects could also be obtained by the addition of Er in Al-Mg series alloys that can not be strengthened by heat treatment ( e.g. the aluminum alloy comprising of Mg4.0 ~ 4.9Wt%, the balance being Al).

In aerospace and aviation industries, the materials used shall be of low specific weight and high elastic modulus, and Al- Li series alloys are regarded as materials to provide with specialty of a low specific weight, and a typical example thereof is the Al-Mg-Li series aluminum alloy 1420 which comprise Mg4.9 ~ 5.5Wt%, Li1.8 ~ 2.1Wt%, Zr0.08 ~ 0.15Wt. However, the alloys still suffer from low

toughness, large anisotropy and insufficient strength. The applicant surprisingly found that the said alloys could be greatly improved in terms of refined grains and increased strength merely by Er alloying.

The present claimed invention is thus fulfilled on the basis of the above discovery.

### 3. Summary of the invention

The present invention aims at providing Er-containing aluminum alloys with greatly improved strength properties without significant deterioration of the toughness.

In a first aspect, the present application provides Al-Zn-Mg-Er and Al-Mg-Er series aluminum alloys. In an embodiment, the Al-Zn-Mg-Er and Al-Mg-Er series aluminum alloys according to the present invention are characterized by comprising 0.1~0.7% of Er (weight percent, and when a composition is addressed hereafter, it is referred to weight percent unless otherwise stated). In a preferred embodiment, the Al-Zn-Mg-Er and Al-Mg-Er series aluminum alloys according to the present invention are characterized by comprising 0.25~0.55% of Er. In a more particular embodiment, the Al-Zn-Mg-Er series aluminum alloys according to the present invention comprise 5.0 ~ 7.0Wt% of Zn, 1.5 ~ 2.5Wt% of Mg and the balance being Al. In a still more particular embodiment, the Al-Zn-Mg-Er aluminum alloys according to the present invention comprises about 6.0% of Zn, about 2.0% of Mg, 0.25~0.55% of Er, and the remainder of Al. In yet a more particular embodiment, the Al-Mg-Er series aluminum alloys according to the present invention comprise 4.0 ~ 5.6Wt% of Mg and the remainder of Al. A preferred Al-Mg-Er series aluminum alloy according to the present invention comprises about 5.0% of Mg, 0.25~0.55% of Er and the remainder of Al.

In another aspect, the present invention provides Er-containing

Al-Li series alloy, i.e. Al-Mg-Li-Zr-Er series alloys. In a preferred embodiment, the Al-Mg-Li-Zr-Er series alloys according to present invention comprise 0.05 ~ 0.70Wt% of Er. In a more preferred embodiment, the Al-Mg-Li-Zr-Er series alloys according to present invention comprise Mg4.9 ~ 5.5Wt%, Li1.8 ~ 2.1Wt%, Zr0.08 ~ 0.15Wt%, Er 0.05 ~ 0.70Wt%, Al being the balance.

Throughout the present description, the composition of each aluminum alloy is regarded that can be modified by the word "about" unless otherwise stated.

The present aluminum alloys can be produced by the following methods:

The conventional melting and ingot casting process was used to produce the Al-Zn-Mg-Er alloys and the Al-Mg-Er alloys, including the following steps: making an Al-Er intermediate alloy from a pure Al metal and a pure Er metal as starting materials by vacuum melting as a first step, and then as a second step melting the Al-Er intermediate alloy together with a pure Al metal and a pure Mg metal (if it is desired to make the present Al-Zn-Mg-Er alloy, a pure Zn metal is further added at this step) in a crucible in an electric resistance furnace and then the resulted melt was poured into a steel mold to produce the desired alloy.

According to the present invention, it is found that the present Er-containing Al-Zn-Mg alloys have a significantly refined grains microstructure, and a dramatically increased strength due to the addition of Er, specifically the tensile strength ( $\sigma_b$ ) and yield strength ( $\sigma_{0.2}$ ) are increased by about 20% with substantially the same elongation property ( $\delta$ ). It is further found that the addition of Er greatly inhibits recrystallization of the Er-containing Al-Zn-Mg alloys, with an increase of recrystallization onset temperature by 50°C

and an increase of recrystallization finish temperature by 80°C. The similar effects are found in Er-containing Al-Mg alloys, specifically the addition of Er contributes to refine the grains of the alloys and increase both the tensile strength ( $\sigma_b$ ) and yield strength ( $\sigma_{0.2}$ ) by about 20%, and increase both the recrystallization onset temperature and the recrystallization finish temperature by 80°C.

Similarly, the conventional melting and ingot casting process was used to produce the Al-Mg-Li-Zr-Er alloys, comprising the following steps: making an Al-Zr intermediate alloy and an Al-Er intermediate alloy from a pure Al metal, a pure Zr metal and a pure Er metal as starting materials by vacuum melting as a first step, and then as a second step melting the Al-Zr intermediate alloy and Al-Er intermediate alloy together with a pure Al metal, a pure Mg metal and a pure Li metal in a carbon coated clay crucible in a vacuum electric resistance furnace and then the gotten melt was poured into a copper mold under an Ar atmosphere to produce the desired Al-Mg-Li-Zr-Er alloy. In a preferred embodiment of the invention, the Al-Mg-Li-Zr-Er alloys are produced by adding element Er in an amount of 0.05 ~ 0.70Wt% into the 1420 alloys. In contrast to the 1420 alloys, the corresponding presently claimed Al-Mg-Li-Zr-Er alloys are characterized by: 1. significantly refined grains, as in fig. 7 (fig. 7 (a) is a metallographic structure picture of the as-cast 1420 alloy; and fig.7 (b) is a metallographic structure picture of the as-cast Al-Mg-Li-Zr-0.2Er alloy); 2. higher degree of age-hardening with an earlier appearance of the peak value as in fig.8 (fig.8(a) is the graph showing the age hardening behavior of the Al-Mg-Li-Zr-0.2Er alloy at 170°C; fig.8(b) is the graph showing the age hardening behavior of the 1420 alloy at 170°C). The strength property of the Al-Mg-Li-Zr-0.2Er alloy reached a peak value under the condition of an ageing time of

mere 6 hours, while it cost 30 hours for the 1420 alloy to reach a peak value; 3. an increase of ageing strength with substantially the same elongation, as in fig. 9, in which it is found that compared with the 1420 alloy, the Al-Mg-Li-Zr-Er alloy enjoys an increase of the tensile strength ( $\sigma_b$ ) by about 7% and an increase of yield strength ( $\sigma_{0.2}$ ) by about 30%, with little change in terms of the elongation ( $\delta$ ).

#### Brief description of the drawings

Fig. 1 is the as-cast metallographic structure of the alloys, in which fig. 1(a) is a picture of an Al-Zn-Mg alloy; fig. 1(b) is a picture of a corresponding Al-Zn-Mg-0.4Er alloy; fig. 1(c) is a picture of an Al-Mg alloy; fig. 1(d) is a picture of a corresponding Al-Mg-0.7Er alloy.

Fig. 2 is a diagram showing the relation between the cold-rolling stretch property and the Er content of the Al-Zn-Mg-Er alloys.

Fig. 3 is a diagram showing the relation between the tensile property and the Er content of the Al-Zn-Mg-Er alloys (quenched at 470°C/0.5h+ aged at 120°C/30h).

Fig. 4 is a diagram showing the relation between the hardness and the annealing temperatures of the Al-Zn-Mg alloy and the Al-Zn-Mg-0.4Er alloy.

Fig. 5 is a diagram showing the relation between the hardness and the annealing temperatures of the Al-Mg alloy and the Al-Mg-0.4Er alloy.

Fig. 6 are pictures of the microstructure of the Al-Zn-Mg alloy and the Al-Zn-Mg-0.4Er alloy annealed at different temperatures, in which fig. 6(a) shows the microstructure of an Al-Zn-Mg alloy (annealed at 325°C/1h); fig. 6(b) shows the microstructure of the corresponding Al-Zn-Mg-0.4Er alloy (annealed at 325°C/1h); fig. 6(c)

shows the microstructure of an Al-Zn-Mg alloy (annealed at 450 °C /1h); fig. 6(d) shows the microstructure of the corresponding Al-Zn-Mg-0.4Er alloy (annealed at 450 °C /1h).

Fig. 7 shows the as-cast microstructure of the alloys (100X), in which fig. 7 (a) is a micrograph of the microstructure of the as-cast 1420 alloy; and fig.7 (b) is a micrograph of the microstructure of the as-cast Al-Mg-Li-Zr-0.2Er alloy.

Fig. 8 shows the age hardening behavior of the alloys at 170 °C , fig.8(a) refers to the Al-Mg-Li-Zr-0.2Er alloy; fig.8(b) refers to the 1420 alloy.

Fig. 9 is a diagram showing the properties of the alloy (quenched at 450 °C /0.5h + aged at 120 °C /12h) varying as a function of the Er contents, tensile strength –  $\sigma_b$ , yield strength –  $\sigma_{0.2}$ , elongation –  $\delta$  .

#### Preferred embodiments of the invention

#### The present Al-Zn-Mg-Er alloys and the present Al-Mg-Er alloys

#### Example 1

An Al-Zn-Mg alloy (1 # alloy of table 1) was produced by the conventional melting and ingot casting process from ultra-high pure Al (with a purity of 99.99%), commercially pure Mg (with a purity of 99.9%) and commercially pure Zn (with a purity of 99.9%) as starting materials. Firstly, 1612.4 grams of the ultra-high pure Al was added and melted in a carbon coated clay crucible in an electric resistance furnace at 780 °C . After the ultra-high pure Al was fully melted, the pure Zn (113.3grams) was further added into the crucible in the electric resistance furnace with thoroughly agitating. Thereafter, the pure Mg (38.6 grams) was added, and after thoroughly agitating 3 grams of hexachloroethane ( $C_2Cl_6$ ) was added for the purpose of degassing and deslagging. Finally, the resulted melt was poured into a

rectangular shaped steel mold, cooled for 3 min. before demolding.

### Example 2

An Al-Zn-Mg-Er alloy (2 # alloy of table 1) was produced by the conventional melting and ingot casting process. An ultra-high pure Al (with a purity of 99.99%) and pure Er (with a purity of 99.9%) as starting materials were melted and cast in a vacuum induce resistance furnace to make an Al-6.2Er intermediate alloy by the blending process (mixture melting process). Then, 27.4 grams of the Al-6.2Er intermediate alloy and 1584.9 grams of the ultra-high pure Al was added into an graphite coated clay crucible, and melted in an electric resistance furnace at 780 °C. After the ultra-high pure Al and the Al-6.2Er intermediate alloy were fully homogeneously melted together, the pure Zn (113.3grams) was further added into the resulted melt with thoroughly agitating. Thereafter, the pure Mg (38.6 grams) was added, and after thoroughly agitating 3 grams of hexachloroethane ( $C_2Cl_6$ ) was added for degassing and deslagging. Finally, the resulted melt was casted into a rectangular shaped steel mold, cooled for 3 min. before demolding. The size of each ingot is  $120 \times 90 \times 32(\text{mm}^3)$ .

### Example 3

The same procedure was repeated as in Example 2 except that the ultra-high pure Al was added in an amount of 1543.7 grams and the Al-6.2Er intermediate alloy was added in an amount of 68.5 grams.

### Example 4

The same procedure was repeated as in Example 2 except that the ultra-high pure Al was added in an amount of 1502.5 grams and the Al-6.2Er intermediate alloy was added in an amount of 109.7 grams.

### Example 5

The same procedure was repeated as in Example 2 except that the ultra-high pure Al was added in an amount of 1461.2 grams and the

Al-6.2Er intermediate alloy was added in an amount of 150.8 grams.

#### Example 6

The same procedure was repeated as in Example 2 except that the ultra-high pure Al was added in an amount of 1420.1 grams and the Al-6.2Er intermediate alloy was added in an amount of 191.9 grams.

#### Example 7

An Al-Mg alloy (7 # alloy of table 1) was produced by the conventional melting and ingot casting process. An ultra-high pure Al (with a purity of 99.99%) and commercial pure Mg (with a purity of 99.9%) were used as starting materials. Firstly the ultra-high pure Al (1665.0grams) was added into a graphite coated clay crucible, and melted in an electric resistance furnace at 780 °C. After the ultra-high pure Al was fully melted, the pure Mg (96.6grams) was further added into the resulted melt with thoroughly agitating. Thereafter, 3 grams of hexachloroethane ( $C_2Cl_6$ ) was added for degassing and deslagging. Finally, the resulted melt was poured into a rectangular shaped steel mold, cooled for 3 min. before demolding. The size of each ingot is 120 ×90 ×32( $mm^3$ ).

#### Example 8

An Al-Mg-Er alloy (8 # alloy of table 1) was produced by the conventional melting and ingot casting process. An ultra-high pure Al (with a purity of 99.99%) and pure Er (with a purity of 99.9%) as starting materials were melted in a vacuum induce resistance furnace and cast to make an Al-6.2Er intermediate alloy by the blending process (mixture melting process). Then, 109.7 grams of the Al-6.2Er intermediate alloy and 1555.0 grams of the ultra-high pure Al was added into a graphite coated clay crucible, and melted in an electric resistance furnace at 780 °C. After the ultra-high pure Al and the Al-6.2Er intermediate alloy were fully melted, the pure Mg (96.6

grams) was added, and after thoroughly agitating 3 grams of hexachloroethane ( $C_2Cl_6$ ) was added for degassing and deslagging. Finally, the resulted melt was poured into a rectangular shaped steel mold, cooled for 3 min. before demolding. The size of each ingot is  $120 \times 90 \times 32(\text{mm}^3)$ .

### Example 9

The same procedure was repeated as in Example 8 except that the ultra-high pure Al was added in an amount of 1472.7 grams and the Al-6.2Er intermediate alloy was added in an amount of 191.9 grams.

The alloys of the above examples were formulated as in table 1.

Table 1 the nominal composition and the amounts of the starting materials(unit: gram).

Note: (1) the total weight of the starting materials for each ingot is 1700g. (2) the melting loss rate: Al - 3%, Mg - 12%, Zn - 10%, the melting loss rate of the intermediate alloy Al-6.2Er is neglected.

No	Nominal compositions of each alloy	Pure Al	Pure Zn	Pure Mg	Al-6.2Er
1 #	Al-6.0Zn-2.0Mg	1612.4	113.3	38.6	0
2 #	Al-6.0Zn-2.0Mg-0.1Er	1584.9	113.3	38.6	27.4
3 #	Al-6.0Zn-2.0Mg-0.25Er	1543.7	113.3	38.6	68.5
4 #	Al-6.0Zn-2.0Mg-0.4Er	1502.5	113.3	38.6	109.7
5 #	Al-6.0Zn-2.0Mg-0.55Er	1461.2	113.3	38.6	150.8
6 #	Al-6.0Zn-2.0Mg-0.7Er	1420.1	113.3	38.6	191.9
7 #	Al-5.0Mg	1665.0	0	96.6	0
8 #	Al-5.0Mg-0.4Er	1555.0	0	96.6	109.7
9 #	Al-5.0Mg-0.7Er	1472.7	0	96.6	191.9

The chemical composition of each ingot was measured by the ICP-AES method, i.e., the inductively coupled plasma atomic emission spectrometry, with a LEEMAN SPEC-E type inductively coupled plasma atomic emission spectrometer. The results are shown in table 2. It is found that the exact compositions are well within the acceptable range of the nominal compositions.

The as-cast samples were taken from the ingots, and observed under a polarized view with a NEOPHOT-21 type optical microscope from Germany. Fig. 1(a) and fig. 1(b) respectively shows the microstructure of the as-cast Al-Zn-Mg alloy and its corresponding Al-Zn-Mg-0.4Er alloy. It is shown from the figures that the microstructure of the as-cast Al-Zn-Mg alloy demonstrates netted structure of coarse dendrite grains, while the Al-Zn-Mg-0.4Er alloy is substantially free from such dendrite grains and featured by refined grains. Fig. 1(c) and fig. 1(d) are the as-cast microstructure of the Al-Mg alloy and its corresponding Al-Mg-0.7Er alloy respectively. It is shown that the microstructure of the as-cast Al-Mg alloy demonstrates very coarse grains, while the Al-Mg-0.7Er alloy is of many refined grains (with about 10 times smaller grain size). It is thus confirmed that the addition of Er significantly refines the as-cast grains of Al-Zn-Mg alloys and Al-Mg alloys.

The ingot was homogenization annealed and then was subjected to hot rolling, intermediate annealing, and cold rolling with a reduction of 60% to yield a sheet of 2mm in thickness. Standard tensile samples were made from the sheet according to Chinese Standard GB6397-86. The samples were tested with a 810MTS(Material Test System) material tester to determine the mechanical properties of the Al-Zn-Mg alloys and the Al-Zn-Mg-Er alloys in both cold-rolled state and quenched and aged state (quenched

at 470 °C /0.5h +aged at 120 °C /30h). The results are shown in fig. 2 and fig.3. The figures show that the addition of Er greatly improve the tensile strength  $\sigma_b$  and yield strength  $\sigma_{0.2}$  of the Al-Zn-Mg alloy with a nominal content of Er of 0.7%, the strength reaches peak value, the cold-rolled Al-Zn-Mg-0.7Er alloy has a tensile strength  $\sigma_b$  of 450Mpa and the quenched and aged Al-Zn-Mg-0.7Er alloy has a tensile strength  $\sigma_b$  of 513Mpa. In contrast, without the addition of Er, the cold-rolled Al-Zn-Mg alloy has a tensile strength  $\sigma_b$  of 350Mpa and the quenched and aged Al-Zn-Mg alloy has a tensile strength  $\sigma_b$  of 420Mpa. However, the Er-containing alloys suffers a reduction of elongation, the cold-rolled Al-Zn-Mg alloy has an elongation of 9%, while cold-rolled Al-Zn-Mg-0.7Er alloy has an elongation of 7%. With adding 0.4% of Er, the Er-containing alloy has a good balance between the quality of strength and the plasticity, the aged Al-Zn-Mg-0.4Er has a tensile strength  $\sigma_b$  of 490Mpa and a elongation  $\delta$  of 10%. Using the same testing process, the tensile strength of the Al-Mg alloy and the Al-Mg-Er alloy in cold-rolled and annealed (at 350 °C /1h) state was shown in table3. From this table, it is found that the addition of Er greatly improves the strength of the Al-Mg alloy. With adding 0.4% of Er, the cold-rolled Er-containing alloy demonstrates an increase by 80Mpa in terms of both the tensile strength  $\sigma_b$  and yield strength  $\sigma_{0.2}$ , and the annealed (at 350 °C /1h) and cold-rolled Er-containing alloy demonstrates an increase by 50Mpa in terms of both the tensile strength  $\sigma_b$  and yield strength  $\sigma_{0.2}$ . With adding 0.7% of Er, the strength of Er-containing Al-Mg alloy undergoes no further increase in strength but a remarkable decrease in elongation: the cold-rolled Al-Zn-Mg alloy has a elongation  $\delta$  of 10.7%, and the Al-Zn-Mg-0.7Er alloy has an elongation  $\delta$  of 5.3%. The results obtained from the examples suggest the preferred range

for the Er addition into the Al-Zn-Mg series alloys and the Al-Mg series alloys varies from 0.25 to 0.55%. Without bounding to any theory, it is presumed that the strengthening effect of Er mainly comes from significant refining of the grains and the formation of various substructures, and Er promotes the precipitation by ageing of reinforcing phases in the Er-containing Al-Zn-Mg alloys, all of which contribute to increase the as-aged strength (fig. 3).

The hardness- metallographic analysis approach was used to determine the recrystallization temperature. Fig. 4 shows the relation between the hardness and the annealing temperatures of the Al-Zn-Mg alloy and the Al-Zn-Mg-0.4Er alloy, and fig. 5 shows the relation between the hardness and the annealing temperatures of the Al-Mg alloy and the Al-Mg-0.4Er alloy. From the two figures, one can primarily determine the recrystallization onset temperature  $T_s$  and the recrystallization finish temperature  $T_f$  (as indicated by the arrows) of the Al-Zn-Mg alloy, the Al-Zn-Mg-0.4Er alloy, the Al-Mg alloy and the Al-Mg-0.4Er alloy. From fig.4, it is found that the recrystallization onset temperature and the recrystallization finish temperature of the Al-Zn-Mg alloy can be increased by about 50°C and by about 80°C respectively, by the addition of 0.4% Er. From fig.5, it is found that the recrystallization onset temperature and the recrystallization finish temperature of the Al-Mg alloy can be both increased by about 80°C, by the addition of 0.4% Er. The microstructure analysis (fig. 6) reveals that after the treatment of annealing at 325°C for 1 hour the recrystallization process had been finished for the Al-Zn-Mg alloy (6(a)), while as for the Al-Zn-Mg-0.4Er alloy the recrystallization process was just beginning (6(b)); after the treatment of annealing at 450°C for 1h the Al-Zn-Mg alloy shows a rather coarse grains (6(c)), while the Al-Zn-Mg-0.4Er alloy shows fine equiaxed grains (6(d)). It

can be thus confirmed that the addition of Er greatly inhibit the recrystallization of the Al-Zn-Mg alloys and of the Al-Mg alloys.

Table 2 the true composition of the alloys (Wt%); \*— alloying element;  
\*\*— true composition

Alloy No.	*	Mg	Zn	Er	Al
**					
1#		1.78	5.28	0	Bal.
2#		1.92	5.73	0.09	Bal.
3#		1.86	5.16	0.24	Bal.
4#		1.71	5.65	0.37	Bal.
5#		1.93	5.70	0.53	Bal.
6#		1.75	5.30	0.62	Bal.
7#		4.41	0	0	Bal.
8#		4.34	0	0.35	Bal.
9#		4.61	0	0.61	Bal.

Table 3 the mechanical properties (tensile) of the Al-5Mg alloy and the Al-5Mg-Er alloys; \*— processing conditions; \*\*— mechanical properties

alloys	As cold rolled			Annealed@350°C /1h		
	$\sigma_b$ (MPa)	$\sigma_{0.2}$ (MPa)	$\delta$ (%)	$\sigma_b$ (MPa)	$\sigma_{0.2}$ (MPa)	$\delta$ (%)
Al-5Mg	352.1	286.5	10.7	283.6	151.9	28.6
Al-5Mg-0.4Er	438.2	367.8	8.6	336.0	205.5	23.8
Al-5Mg-0.7Er	435.8	363.4	5.3	338.7	207.9	19.3

### The present Al-Mg-Li-Zr-Er alloys

The 1420 alloy and the Al-Mg-Li-Zr-Er alloy were formulated and melted, based on a volume weight of 10 kg of the cast mold. The alloying elements Zr and Er were added in the form of the Zr-Al and Er-Al intermediate alloy. The starting materials for Al, Mg, Li were of high purity metals. The melting process was conducted under Ar atmosphere in a ZGG-0.025 vacuum induction furnace. It is to be noted that the intermediate alloys were made in the vacuum furnace by the blending process from high purity Al, high purity Zr and high purity Er as starting materials, to yield an Al-3.6wt% Zr alloy and an Al-6.2wt% Er intermediate alloy.

#### Example 10 (comparative example)

A 1420 alloy (10 # alloy of table 4) was formulated and melted by the conventional melting and ingot casting process from ultra-high pure Al (with a purity of 99.99%), ultra-high pure Mg (with a purity of 99.99%) and ultra-high pure Li (with a purity of 99.99%) and intermediate alloy Al-3.6wt% Zr, based on a volume weight of 10 kg of the cast mold. 8899 grams of the ultra-high pure Al and 361 grams of the Al-Zr intermediate alloy were added into an alumina crucible, and then put into the ZGG-0.025 vacuum induction furnace. When the pressure inside the furnace was lower than 0.3Pa, electricity was conducted to heat and melt all the materials, and then was shut down when the melt reached a temperature of 760°C, and vacuum was kept for degassing. When the temperature of the melt was lowered to 710°C and the vacuum degree is higher than 0.3Pa, 200mmHg Ar gas was flowed into the furnace, and 540 grams Mg and 200 grams of Li (well packaged with Al foil) were added. Electricity was pulse conducted, and the melt was agitated intensively, and then the melt was poured into water-cooled copper mold when the temperature raised to 740°C ~ 760°C. The size of each ingot was of 290 × 200 × 70m.

### Example 11

An Al-Mg-Li-Zr-0.05Er alloy (11 # alloy of table 4) was formulated and melted by the conventional melting and ingot casting process from ultra-high pure Al (with a purity of 99.99%), ultra-high pure Mg (with a purity of 99.99%) and ultra-high pure Li (with a purity of 99.99%) and the intermediate alloy Al-3.6wt % Zr and Al-6.2wt % Er, based on a volume weight of 10 kg of the cast mold. 8818 grams of the ultra-high pure Al, 361 grams of the Al-Zr intermediate alloy and 81 grams of the Al-6.2Er intermediate alloy were added into an alumina crucible, and then put into the ZGG-0.025 vacuum induction furnace. When the pressure inside the furnace was lower than 0.3Pa, electricity was conducted to heat and melt the materials, and was shut down when the melt reached a temperature of 760°C, and vacuum was kept for degassing. When the temperature of the melt was lowered to 710°C and the vacuum degree is higher than 0.3Pa, 200mmHg Ar gas was flowed into the furnace, and 540 grams Mg and 200 grams of Li (well packaged with Al foil) were added. Electricity of the furnace was pulse conducted, and the melt was agitated with a high power, when the temperature raised to 740°C ~ 760°C the melt was poured into a water cooled copper mold. The size of each ingot was of 290 × 200 × 70m.

### Example 12

The same procedure was repeated as in Example 11 except that the ultra-high pure Al was added in an amount of 8737 grams and the Al-6.2Er intermediate alloy was added in an amount of 162 grams.

### Example 13

The same procedure was repeated as in Example 11 except that the ultra-high pure Al was added in an amount of 8575 grams and the Al-6.2Er intermediate alloy was added in an amount of 324grams.

#### Example 14

The same procedure was repeated as in Example 11 except that the ultra-high pure Al was added in an amount of 8332grams and the Al-6.2Er intermediate alloy was added in an amount of 567 grams.

#### Example 15

The same procedure was repeated as in Example 11 except that the ultra-high pure Al was added in an amount of 8008 grams and the Al-6.2Er intermediate alloy was added in an amount of 891 grams.

#### Example 16

The same procedure was repeated as in Example 11 except that the ultra-high pure Al was added in an amount of 7765 grams and the Al-6.2Er intermediate alloy was added in an amount of 1134 grams.

The chemical composition of each ingot was measured by the ICP-AES method, i.e., the inductively coupled plasma atomic emission spectrometry, with a LEEMAN SPEC-E type inductively coupled plasma atomic emission spectrometer. The results are shown in table 4.

The as-cast samples were taken from the ingots, and observed with a OLYMPUS-PMG3 type optical microscope. Fig. 7(a) and fig. 7(b) respectively shows the as-cast microstructure of the 1420 alloy and its corresponding Al-Mg-Li-Zr-Er alloy. It is shown from the figure 7 that the microstructure of the as-cast Al-Mg-Li-Zr-0.2Er alloy shows a much finer structure than that of the 1420 alloy.

The hardness was measured with a HBWUV-187.5 optical Brinell hardness tester, according to the Rockwell hardness testing method (by the steel ball of a diameter of 1.588mm, with a load of 980N). The hardness of aged samples (solid solution treated at 450°C ×30min, and aged at 170°C for different time) is shown as fig. 8. The figure 8 shows that the addition of Er increases the hardness of the aged

Er-containing alloy and promotes earlier appearance of the ageing peak value.

The ingot was homogenization annealed and then was subjected to hot rolling, intermediate annealing and cold rolling with a reduction of 50% to yield a sheet of 2mm in thickness. Standard tensile samples were made from the sheet according to Chinese Standard GB6397-86. The samples were tested with a 810MTS(Material Test System) material tester to determine the mechanical properties of the alloys in aged state (solid solution treated at  $450^{\circ}\text{C} \times 30\text{min}$ , and aged at  $120^{\circ}\text{C}$  for 12 hours). The results are shown in fig. 9. The figure shows that the addition of Er greatly improve the yield strength  $\sigma_{0.2}$  of the 1420 alloy. With Er content ranging from 0.05 to 0.70wt%, the strength of the Al-Mg-Li-Zr-Er is higher than that of its counterpart 1420 alloy (the yield strength  $\sigma_{0.2}$  of the 1420 alloy is 233.79MPa). With an Er content of 0.55%, the Er-containing alloy reaches a maximum of yield strength of 300.05Mpa. Compared with the 1420 alloy, the Al-Mg-Li-Zr-Er exhibits a comparable plasticity. The elongation  $\delta$  of the 1420 alloy is 19.09%, while the elongation of each of the Al-Mg-Li-Zr-Er alloy is no less than 15.4%, among the alloy system the Al-Mg-Li-Zr-0.2Er alloy is the highest (17%).Without bounding to any theory, it is presumed that the strengthening effect of Er mainly comes from the significant refining of the grains and the formation of various substructures, and Er promotes the precipitation by ageing of reinforcing phases in the Er-containing Al-Mg-Li-Zr alloys, all of which contribute to greatly increase the as-aged strength (fig. 9).

Table 4 the chemical composition and the mechanical properties of the alloys

Sample No	Composition (Wt%)					Mechanical properties		
	Mg	Li	Zr	Er	Al	Tensile strength $\sigma_b$ (Mpa)	Yield strength $\sigma_{0.2}$ (Mpa)	elongation $\delta$ (%)
Ex.10 (Comp.)	5.20	1.97	0.12	0	bal.	440.49	233.79	19.09
Ex.11	5.05	1.91	0.08	0.05	bal.	457.39	266.65	17.58
Ex.12	5.50	1.88	0.12	0.10	bal.	454.9	269.28	17.2
Ex.13	4.96	1.94	0.10	0.20	bal.	467.72	292.04	17
Ex.14	5.30	1.86	0.13	0.35	bal.	467.12	297.02	16.4
Ex.15	5.35	2.08	0.13	0.55	bal.	470.24	300.05	16.4
Ex.16	4.90	1.83	0.15	0.70	bal.	458.34	272.58	15.4